

# PATENT SPECIFICATION

(11)

1 509 680

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- (21) Application No. 39799/76 (22) Filed 24 Sept. 1976 (19)  
(31) Convention Application No. 4576/75 (32) Filed 10 Oct. 1975 in  
(33) Denmark (DK)  
(44) Complete Specification published 4 May 1978  
(51) INT. CL.<sup>3</sup> C01B 25/32  
(52) Index at acceptance  
C1A D37 G47 G47D37



## (54) A PROCESS FOR PRODUCING A POWERED OR GRANULATED NON-DUSTING MINERAL COMPOSITION

(71) We, DANSK LANDBRUGS GROV-  
VARESELSKAB AXELBORG, a Danish Com-  
pany of DK-1503 Kobenhavn V, Denmark,  
do hereby declare the invention, for which  
5 we pray that a patent may be granted to us,  
and the method by which it is to be per-  
formed, to be particularly described in and  
by the following statement:—

This invention relates to a process for  
10 producing a powdered or granulated, non-  
flocculating mineral composition containing

known to react a preheated aqueous suspen-  
sion of calcium carbonate with preheated  
phosphoric acid under conditions producing  
continuously a foamed material, which is  
distributed in a gas wherein the reaction  
product completes the reaction and is dried  
in freely suspended state to form a powder. 55

It is the object of the present invention  
to teach a simplified procedure for non-  
dusting calcium phosphate blends, such as  
di- and monocalcium phosphate, and 60

## ERRATA

### SPECIFICATION NO 1509680

Page 1, line 2, *after VARESELSKAB delete AXELBORG*

Page 1, line 3, *after of insert Axelborg*

THE PATENT OFFICE  
20 June 1978

Bas 49145/8

## ERRATUM

Slip No. 2

### SPECIFICATION NO 1509680

Page 1, Heading (54) for POWERED *read* POWDERED

THE PATENT OFFICE  
12 December 1978

Bas 52589/6

3,391,992 it is known to prepare dicalcium  
45 phosphate for use as fodder by reacting  
diluted superphosphoric acid and a calcium  
salt such as calcium carbonate. This pro-  
cess requires hydrolysis of the superphos-  
phoric acid as a first step and proceeds dur-  
ing intensive development of heat.

50 For Norwegian Patent No. 132,685 it is

ate in the form of ground chalk having a  
content of water of from 15 to 20 percent  
by weight, and the phosphoric acid is pre-  
ferably an ordinary commercial phosphoric  
acid of a concentration corresponding to  
50—55 percent by weight of P<sub>2</sub>O<sub>5</sub>. 95

The process according to the invention  
will be illustrated below by way of examples. 100

SEE ERRATA SLIP ATTACHED

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## (54) A PROCESS FOR PRODUCING A POWERED OR GRANULATED NON-DUSTING MINERAL COMPOSITION

(71) We, DANSK LANDBRUGS GROV-VARESELSKAB AXELBORG, a Danish Company of DK-1503 Kobenhavn V, Denmark, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

10 This invention relates to a process for producing a powdered or granulated, non-flocculating mineral composition containing calcium phosphates, preferably for use as fodder, and wherein aqueous phosphoric acid is reacted with calcium carbonate.

15 Calcium phosphates, such as dicalcium phosphate, monocalcium phosphate and mixtures thereof, are used extensively as components in mixed feeds or feed supplements for the feeding of domestic animals.

20 The said salts can be used alone or admixed with other vital minerals such as various metal compounds, for instance salts of iron, zinc, manganese, copper and cobalt, and, if desired, vitamins and other additives.

25 Such mineral compositions are normally mixed in the fodder or drinking water in prescribed ratios, so that the mineral requirement of the animals will be covered.

Mineral compositions or feed supplements 30 having a high content of mineral compounds are normally prepared by blending the dry components in the desired ratio. Of the drawbacks attending this procedure may be mentioned that the prepared mixtures will normally be dust-containing which is unpleasant or even dangerous to both humans and animals, because the fine particles may penetrate into the lungs. Besides, it is difficult in the conventional procedure 35 to obtain an adequately uniform distribution of all the components in the mixture.

From the specification of US Patent No. 3,391,992 it is known to prepare dicalcium phosphate for use as fodder by reacting 40 diluted superphosphoric acid and a calcium salt such as calcium carbonate. This process requires hydrolysis of the superphosphoric acid as a first step and proceeds during intensive development of heat.

45 For Norwegian Patent No. 132,685 it is

known to react a preheated aqueous suspension of calcium carbonate with preheated phosphoric acid under conditions producing continuously a foamed material, which is distributed in a gas wherein the reaction product completes the reaction and is dried in freely suspended state to form a powder.

55 It is the object of the present invention to teach a simplified procedure for non-dusting calcium phosphate blends, such as di- and monocalcium phosphate, and mineral blends containing these salts.

The process according to the invention is characterized by the mixing of aqueous phosphoric acid of a concentration of at least 40 percent by weight of P<sub>2</sub>O<sub>5</sub> with a finely divided calcium carbonate having a content of water of from 15 to 25 percent by weight, whereafter other desired minerals and vitamins are admixed and the mixture, if desired, is subsequently dried. In this process the reaction between phosphoric acid and calcium carbonate is effected rapidly and smoothly in batches and proceeds to completion while forming a uniform reaction product. This is a result of the presence of a substantial content of moisture in the calcium carbonate, which ensures a satisfactory and uniform distribution of the phosphoric acid in the reaction mix. The finished mixtures will be non-dusty, and also in cases where other mineral components and/or vitamins or other additives are added. If the process is combined with a subsequent drying step after all the supplementary components have been added to the reaction mass, the result will be a certain agglomeration whereby fine particles present in the mass will adhere to form a dry non-dusting granulate or powder.

80 It has been found to be expedient according to the invention to use calcium carbonate in the form of ground chalk having a content of water of from 15 to 20 percent by weight, and the phosphoric acid is preferably an ordinary commercial phosphoric acid of a concentration corresponding to 50—55 percent by weight of P<sub>2</sub>O<sub>5</sub>.

85 The process according to the invention will be illustrated below by way of examples. 100

**SEE ERRATA SLIP ATTACHED**

**EXAMPLE 1**

While stirring vigorously 76.1 kg powdered white chalk having a content of water of 17.0% and a content of  $\text{CaCO}_3$  of 76.4%, 5 corresponding to 92.0%  $\text{CaCO}_3$  of dry components, was mixed with 76.7 kg phosphoric acid solution containing 53.8%  $\text{P}_2\text{O}_5$ , or 74.3%  $\text{H}_3\text{PO}_4$ , and 25.7% of water.

During the mixture some matter evaporated, and the result was 127.2 kg solid matter with 22.1 kg free water. The end product after drying was 105.1 kg of dry non-dusting granulate containing 5.1 kg clay and/or sand and 100 kg  $\text{Ca(H}_3\text{PO}_4)_2 \cdot 2\text{H}_2\text{O}$ .

**EXAMPLE 2**

The procedure of Example 1 was repeated using 52.0 kg powdered white chalk and 104.7 phosphoric acid of the composition specified above. The result was 139.2 kg solid matter with 35.7 kg free water, which partly evaporates. The product was dried to a granulate weighing 103.5 kg and containing 100 kg  $\text{Ca}(\text{H}_3\text{PO}_4)_2 \cdot 2\text{H}_2\text{O}$ .

**WHAT WE CLAIM IS:—**

1. A process for producing a powdered or granulated, non-dusting mineral composition containing calcium phosphates, preferably for use as fodder, including the step of 20 mixing aqueous phosphoric acid of a concentration of at least 40 percent by weight of

$\text{P}_2\text{O}_5$ , with a finely divided calcium carbonate having a content of water of from 15 to 25 percent by weight to react therewith.

2. A method according to claim 1, 35 wherein the finely divided calcium carbonate is ground chalk having a content of water of from 15 to 20 percent by weight.

3. A method according to claim 1 or 2 wherein said phosphoric acid is phosphoric acid of a concentration corresponding to 40 50—55 percent by weight of  $\text{P}_2\text{O}_5$ .

4. A method according to any preceding claim including the subsequent step of admixing other minerals and/or vitamins.

5. A method according to any preceding claim including the step of drying the reaction product of the method of any one of claims 1 to 3 or the mixture prepared by the method of claim 4.

6. A product manufactured by the method of any preceding claim.

7. A method for producing powdered or granulated non-dusting mineral composition containing calcium phosphates as claimed in claim 1 substantially as herein described,

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